TECHNICAL REPORT

HIGH TEMPERATURE THERMAL DIFFUSIVITY FURNACES AND TECHNIQUES FOR MEASUREMENT

BY

W. B. Crandall and G. E. Potter

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Alfred University

Alfred, New York

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ABSTRACT

A low temperature (200° - $1400^{\circ}C$) and a high temperature (1000° - $2000^{\circ}C$) thermal diffusivity apparatus is described. The high temperature apparatus incorporates a low temperature as well as a high temperature furnace, so that it is possible to measure two sets of thermal constants, one at each temperature all with the same set-up. In addition to thermal diffusivity, this apparatus will measure Biot's modulus and surface heat transfer coefficient.

Introduction

The development of new power plants for high speed aircraft and other types of propulsion equipment has made the measurement of thermal constants at high temperatures increasingly important. Probably of foremost interest in the field of high temperature constants is that of thermal conductivity or thermal diffusivity. A knowledge of exactly how heat is distributed through construction materials for these power plants is essential, for without this knowledge it is impossible to construct components which will be compatable throughout regarding heat distribution.

Unsteady state methods for measuring thermal constants offer several advantages over the steady state methods. Consequently an unsteady method was devised for measuring thermal constants over a wide temperature range. This method has been described by H. S. Levine[1]. It is this method which has been employed for measuring thermal diffusivity over two ranges. The first from approximately 200 to 1400°C and the second from 1000 to 2000°C.

One of the principle difficulties in obtaining reliable data from this method has been that of obtaining the exact experimental conditions postulated by the theory. The furnaces described in this paper are the outcome of an investigation concerning the proper manner of meeting the boundary condtions postulated.

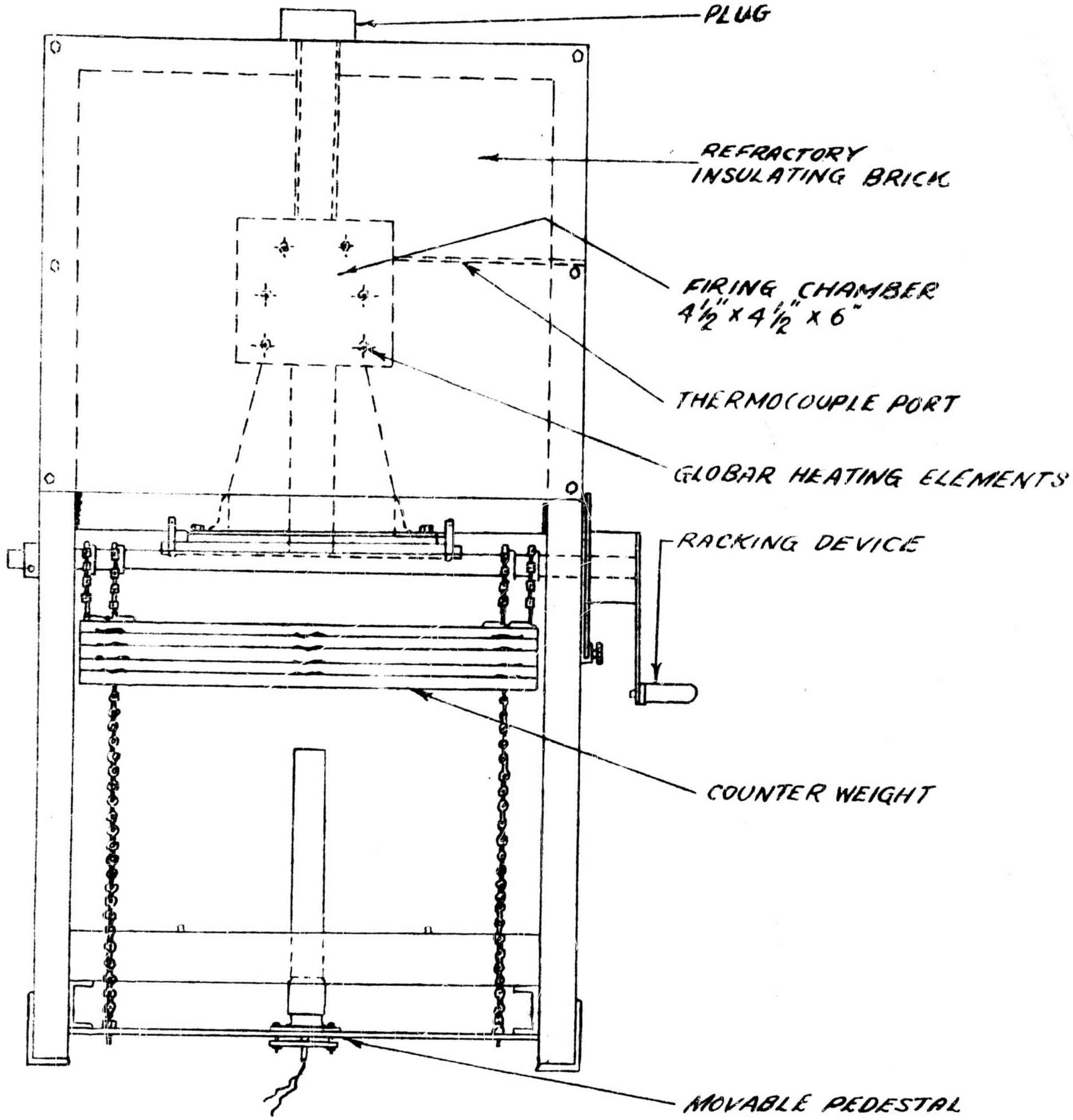
Furnace Construction

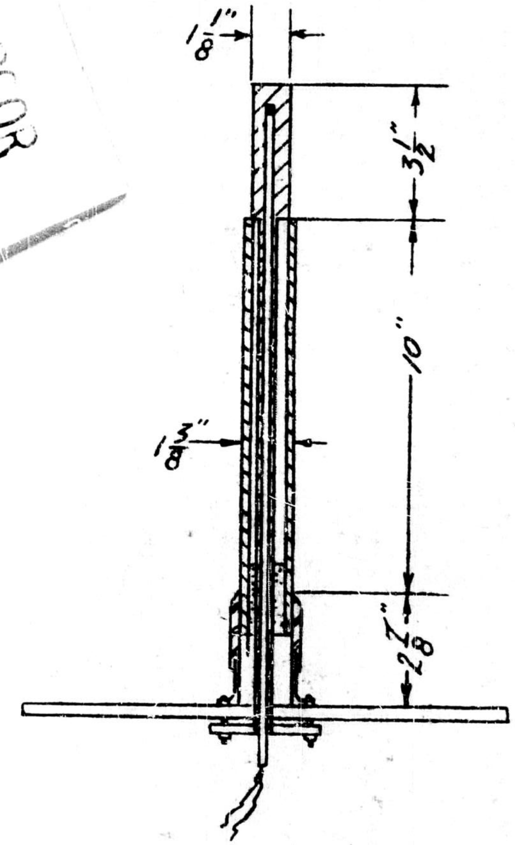
The first thermal diffusivity apparatus was used for the lower temperature range, i.e., 200 - 1400°C. All of the first experiments were made on this furnace with a view towards possible improvements both on this unit and a later model which could be used at higher temperatures. The second thermal diffusivity apparatus was constructed incorporating the above mentioned improvements but with the same general principle as the first.

I. Construction of the Low Temperature Thermal Diffusivity Apparatus.

Figure 1 indicates the furnace assembly used for the low temperature thermal constants measurements. This furnace was briefly described by H. S. Levine[1]. As the drawing indicates, it is a Harper Electric Globar furnace which was converted for this special application. As received this inverted pit type furnace had a removable hearth. That is, the racking device could be used to remove the entire bottom of the furnace to facilitate the loading and unloading of bulky material. A new hearth was constructed exactly similar to the original except with a hole 1 7/16" in diameter along its center axis. This hearth was bolted into permanent position blocking off the bottom of the furnace. A movable pedestal was replaced on the racking device in such a position that the pedestal could be moved through the hole in the hearth to the desired position by simple cranking of the racking device. Also as received this furnace had six globars, three on each side stacked vertically. After exploration of the interior of this furnace under heating conditions it was found desirable to move the top two globars inward toward the center line of the furnace approximately 1 1/2" to facilitate even distribution of heat throughout the chamber.

Measurement of thermal constants have been made using this device up to 1400°C, and could possibly be used successfully up to 1500°C. However, in view of the fact that data were desired at temperatures well in excess of even 1500°C it was felt that an entirely new set-up should be devised which would allow for the higher temperature measurements as well. The new set-up was to incorporate several improvements, making the device more convenient to operate as well as perhaps meeting the boundary condition somewhat better.





MOVABLE PEDESTAL
(WITH SPECIMEN MOUNTED)

FIG. 1 - FURNACE E-1

HARPER ELECTRIC FURNACE CONVERTED FOR THERMAL DIFFUSIVITY MEASUREMENTS
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II. Construction of the High Temperature Thermal Diffusivity Apparatus.

Levine's unsteady state method of measuring thermal constants becomes more precise the greater the temperature difference between the starting and final temperature, if all of the boundary conditions are satisfied. However, as this temperature difference becomes greater the difficulties involved in making the measurements of temperature rise on the thermocouple become greater. Also, as the temperature difference becomes greater so also does the thermal shock on the specimen and other parts of the system being plunged into the hot medium. For this reason two furnaces were required in the set-up, one which could be operated at an intermediate temperature as well as one for high temperature.

Upon exploring the possibilities of construction of these two furnaces many factors required consideration, especially as they concerned meeting with the proper postulated boundary conditions. They were:

Relative position of the furnaces,

Effective size and shape of the heating chambers,

Heating systems to be used in furnaces,

Effect of protective atmosphere, if necessary,

Method of quickly plunging the specimen first into one furnace then into the other,

Temperature measuring device in furnaces and inside the specimen suitable for this temperature range,

Relative expense of the unit.

<u>Relative Position of the Furnaces</u> - The original set-up, described in the earlier section, was constructed so that the specimen could be moved upward into the furnace chamber. This system was found to have many advantages; such as, being able to keep the specimen (in the shape of a semi-infinite cylinder) upright on the pedestal with only the thermocouple holding it in place, that is, requiring no cementing, and

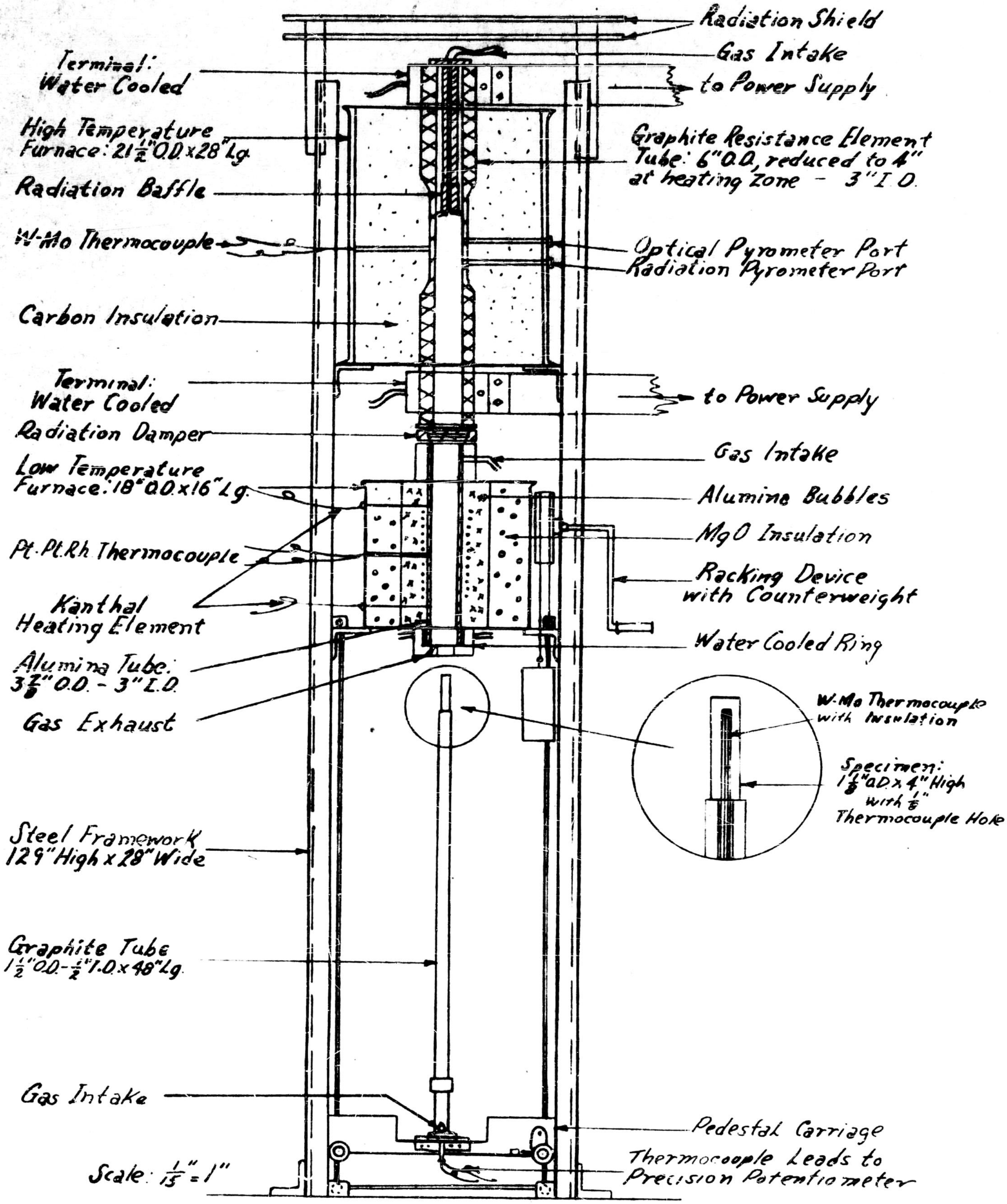

Fig. 2 HIGH TEMPERATURE THERMAL DIFFUSIVITY APPARATUS

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keeping the specimen at a uniform temperature prior to entry into the furnace. With these features in mind it was considered advisable to construct the second device in a similar manner. This situation required that the lower temperature furnace be at the bottom and the higher temperature furnace at the top separated by as small a distance as possible so that no temperature drop would be obtained in passing between the furnace. Figure 2 illustrates the high temperature thermal diffusivity apparatus.

Effective Size and Shape of the Heating Chamber - The size and shape of the specimen required consideration first before the dimensions of the heating chamber could be established. Again it was necessary to draw on information from the first unit for this consideration. A specimen 1 1/8" in diameter by 3 1/2 to 4" long had been used in the first measurements. This size and shape specimen had been found to obey the conditions of the semi-infinite cylinder when supported on a pedestal from below, the pedestal being used in part to extend the specimen in the one infinite direction. A consideration was given to the heat conduction equation for other regularly shaped specimens, and it was found that the heating boundary conditions could probably best be satisfied using this same size and shaped specimen if a cylindrically shaped heating chamber of the right dimensions and heat capacity was used. For this reason, tublar furnaces were constructed having a three inch inside diameters and 10 to 12 inch heating lengths.

Heating System to be Used in Furnaces - After the desirable size and shape of furnaces was determined it was necessary to obtain the proper heating system for these chambers. The lower temperature furnace required very little consideration. The temperature required in this furnace would be relatively low, approximately 1100°C maximum. A wire wound tube furnace was constructed using number 15 gauge Kanthal A-1 wire. The wire was wound on a Norton RA 164 tube with 3" I.D., 3 7/8" O.D. and 21" long. The wire was cemented in place using RA 563 Norton cement.

This element was heated using a simple auto type transformer. The heating element was surrounded by a 2" layer of alumina bubbles and these surround by (light powdered) MgO insulation, the whole being contained in a 16 gauge stainless steel drum, 18" O.D. and 16" long. Attached to the bottom of this furnace is a water cooled ring with a gas exhaust used for cooling the gas seal. Attached to the top of the furnace is a gas intake system.

The upper, high temperature, furnace required much more consideration as this was to be operated above the normal heating range. One of the simplest manners of obtaining a cylindrical heating chamber is to make the heating element itself in this shape. Upon considering the heating elements which could be used in the 1800 - 2000°C + range and which might be used to obtain this shape of heating chamber, it seemed that either carbon or graphite was the logical solution, if the proper protective atmosphere could be tolerated. Also entering this consideration comes other things such as, the distribution of heat in the control zone, the power necessary to heat such a chamber, the manner of delivering this power to the heating elements, the control of temperature and the influence of possible carbon fumes on the experiment. After considering these problems the following solution was obtained. A graphite tube was shaped so that a uniform heating chamber could be obtained in the center at least 4 - 6" long using a total of 12" of heating length, this tube to be heated by electrical resistance. An extruded graphite cylinder 40" long was obtained having a 3" hole drilled through the center and 6 1/8" O.D. The ends were machined to exactly 6" diameter to fit the bronze terminals accurately. The purpose being that there should be intimate contact with the terminals to insure good electrical contact with the transformer leads. The 12" section in the middle of the resistor was turned to a 4" diameter making a higher resistance in this section for the heating zone. This section was turned with a large radius to maintain as much strength as possible. From the resistivity of this graphite tube the transformer power supply specifications were drawn. A transformer was obtained having the following specifications:

50 KVA

230 Volt primary

0-10 Volt secondary in 2 volt steps

5000 amps maximum secondary

10 Volts maximum secondary

This amount of current required a rather sizable lead to the furnace. In addition, the lead needed to allow for expansion of the graphite tube during heating. For this purpose a laminated lead was constructed using thirty-five, 4 1/4" x 0.021" (24 gauge) or copper straps fastened together. The terminals used to connect the transformer leads to the graphite resistor were made of bronze castings because of the high conductivity of the copper in the bronze. The casting was split and two pieces brazed into position at the split to provide a means of clamping onto the graphite resistor. A brass strap was brazed around the outside to provide a closed space in the terminal for cooling water to run. When the casting was bored to the 6" diameter, the castings proved to be porous. To eliminate the leaks 24 gauge copper stock was sweated inside the terminal.

To provide a more even heat distribution to the top surface of the specimen, a combination gas part and radiation baffle of graphite was placed in the top portion of the heating zone. The thin graphite baffle when heated served to distribute the heat more evenly to the top of the specimen. The radiation baffle support was necked down in the middle so that heat would not be conducted away too rapidly.

Around the graphite resistor was placed powdered carbon insulation. The whole was contained in a 16 gauge stainless steel drum with convenience openings to load and unload the carbon insulation.

The graphite resistor when the furnace is cold, hangs from the clamped top terminal. The resistor expands during heating so that the lower end rests on the top of the radiation damper and any further expansion just causes the upper terminal to rise. This was done so that there wouldn't be an excessive loading on the lower furnace assembly all of the time still a rather tight seal would be accomplished during the testing period.

Effect of Protective Atmosphere, if Necessary - Protective atmospheres must be used if oxidation of the graphite parts is to be eliminated. This requires that a rather tight system must be maintained at all times during operation. A seal is maintained at these points throughout the system: At the top of the upper furnace by a graphite washer, at the bottom of the top furnace by a machined transite washer between the graphite tube and the machined top of the radiation damper, at the bottom of the radiation damper by a machined fit between the damper and the top of the lower furnace collar, (Another gas intake is provided at this point to insure sufficient protection to all of the parts in the lower furnace), and at the bottom of the bottom furnace by a stopper which is fitted around the specimen support and into the water cooled ring: A gas exhaust is supplied in the water cooled ring also. The upper and lower furnace tubes will contain the protective atmosphere if an ample flow is provided as they are sufficiently non-porous at their operating temperatures.

Method of Quickly Plunging the Specimen First Into One Furnace Then into Other -

The support which carries the specimen into the furnaces must be capable of withstanding all of the conditions of both furnaces. For this reason the support was also made of graphite, for this was the only practical material which would withstand all conditions, including the very sever thermal shock and high temperatures of the upper furnace. This however, did necessitate placing a protective atmosphere in the lower furnace. Because of the need for an protective atmosphere in the lower furnace and because of the very high radiation affect from the upper furnace a radiation damper was placed between the two furnaces. This damper was provided with a sliding (mashined fitted) steel plate in it with a 3" hole in one end and solid on the other so that quick access to the upper furnace could be obtained.

The graphite specimen support is provided with a hollow center so that the thermocouple and protective insulation may be passed through it. The entire tube assembly is carried on a pedestal carriage. The carriage and racking device for the carriage is mounted on the same framework as the two furnaces. The framework provides a track for the carriage rollers and ensures that the pedestal can be moved along the central axes of the two furnaces. The carriage is counter-weighed over the racking device so that the assembly may be moved up and down easily and will remain at any set postion. A gas intake is provided at the base of the graphite tube specimen support to supply a protective atmosphere to the inside of the tube and to the thermocouple. A gas seal is maintained at all points surrounding the base of this tube below the point at which the gas is introduced.

<u>Temperature Measuring Devices in Furnace and Inside the Specimen, Suitable for This Temperature Range</u> - Probably the most convenient method of measuring temperatures in the 1800 - 2000°C range is by the use of an optical or radiation pyrometer. Because the temperature must be known at a point within the specimen at such a great distance from the outside of the furnace these methods are not suitable. A thermocouple is the logical solution to this problem if one may be used which will measure the high temperature and still not be "lost" during the run. There are several metal combinations that may be used in this range, however, everything considered the tungsten - molybdenum thermocouple combination was the one selected for use. Although this metal combination does not have a high e.m.f. - temperature coefficient and in fact does not even have a position coefficient at all temperatures, and requires a protective atmosphere during heating, it may be calibrated and used satisfactorily in this application[2]. Also the lower cost of these metals

allowes one to connect the couple directly to the measuring means and to replace the thermocouples frequently.

The selection of a thermocouple always brings with it the selection of the insulating material to be used with it. Again the common materials are not designed for this elevated temperature and a selection must be made considering not only the usable temperature range of the material, but the reactivity of the thermocouple and the specimen with that material at the elevated temperature. For the tests to be conducted in the temperature range up to 1950°C a very pure alumina was chosen. Both the temperature requirements and reactivity was satisfactory if the insulator was changed frequently. The Morganite Company of England was able to supply these insulators at a cost which allowed frequent changes to be made. Stabilized zirconium oxide insulators may be used above 1950°C, however, fabrication of this material in a shape small enough for this application in quite difficult (size, - 0.125 O.D. and double bore 0.03" bore).

Temperature measurement and control in the furnaces presents a somewhat different situation than in the specimen for here it is possible to place sighting tubes directly on the desired area. For this reason, all three types of temperature measuring devices were provided for in the upper furnace; optical pyrometer, radiation pyrometer and tungsten-molybdenum thermocouple, so that the merrits of each might be determined and the proper means selected. In the lower furnace a platinum - platinum rhodium thermocouple was used as the measuring device. The control of temperature in both furnaces was obtained by manual adjustment of power input. Fairly long periods of time were required to stabilize the temperature in these furnaces so that equilibrium temperature could be maintained prior to entry of the specimen.

<u>Relative Expense of the Unit</u> - Into the design and construction of this unit went the following components of cost:

1. Engineering time - 14 man days

2. Labor time - 50 " "

3. Material Costs -

   A. Furnace assembly minus temperature measuring devices = $700.00

   B. Power transformers - $1800.00

Operating Techniques

A discussion of the operation of these apparatuses will be confined to the high temperature thermal diffusivity set-up for in this device is embodied all of the techniques involved in the operation of both units. The high temperature apparatus is operated in the following manner:

I. Establishment of temperature in furnaces.

   A. Upper furnace -

      1. Start water through water cooled terminals.

      2. Start neutral gas flowing into furnace thru gas intake at top of furnace.

      3. Close radiation damper between furnaces.

      4. Turn on power to furnace.

      5. Observe hot chamber with optical pyrometer and bring temperature up to desired operational point by adjusting the power input.

      6. Allow furnace to come to equilibrium temperature conditions.

NOTE: When the W-Mo thermocouple is used, a neutral gas is supplied to the couple at all times.

   B. Lower furnace -

      1. Start water through lower water cooled ring.

      2. Block off sample entrance port at bottom of furnace.

3. Determine temperature of the furnace with temperature measuring device on platinum - platinum rhodium thermocouple and bring temperature up to desired operational point by adjusting the power input. (The small temperature difference between the temperature of this thermocouple and the temperature of the interior has previously been determined and this difference allowed for).

4. Allow furnace to come to equilibrium temperature condition.

II. Preparation of specimen on support.

A. Place previously annealed and calibrated W-Mo thermocouple inside graphite support tube with wires attached to precision potentiometer.

B. Position the specimen on the thermocouple end which protrudes from the graphite tube so that when the bare bead of the thermocouple is just touching the bottom of the hole in the specimen, the base of the specimen is resting on the top of the graphite support tube. (See insert on Figure 2).

C. Set screws at the bottom of the graphite tube support may then be tightened thus fixing the position of the thermocouple.

D. A neutral gas is introduced into the gas intake port at the base of the graphite support tube, and "Plastisene" is placed around the point at which the thermocouple wires leave this tube to seal off the gas flow.

E. The temperature of the specimen is recorded and the e.m.f. of the thermocouple in the specimen is read.

III. Measurement of thermal constants..

A. A neutral gas is started through the lower furnace and the block removed from the specimen entrance port.

B. The racking device is used to position the specimen in the temperature control zone of the lower furnace, and the sliding stopper positioned in the water cooled ring to seal off the bottom of the furnace.

C. Zero time is indicated as the specimen reaches this position. The time is then recorded for that point in the specimen to reach certain prearranged e.m.f.'s (temperatures). These e.m.f.'s having been selected for each specimen so that the operator of the precision potentiometer is physically able to make the temperature readings accurately. The temperature coefficient of e.m.f. is both negative and positive in the temperature range below 1100°C thus requiring a potentiometer which will make these readings possible with the greatest speed and convenience possible. It is necessary to supply additional power to the furnace during this operation to maintain a constant temperature in the control zone during the test run.

D. After the specimen has reached the equilibrium temperature of the lower furnace the movement to the upper furnace may be made. The radiation damper is moved to clear the opening and the racking device used to position the specimen in the controlled temperature zone of the upper furnace. The sliding seal at the bottom of the lower furnace fixes the seal of the system.

E. Zero time is indicated as the specimen reached this position. The same procedure is then followed as for the lower temperature furnace and the times to reach prearranged temperatures recorded.

F. After the specimen has reached the equilibrium temperature of the upper furnace the test run is completed and the racking device may be used to remove the specimen from the furnaces as quickly as the thermal shock resistance of the material will permit. The furnace and the neutral gas may be turned off as the temperature permits.

It is necessary to establish three heating curves obtained in the above described manner, each curve being obtained using a different position of the thermocouple in the specimen. From these heating curves it is possible to make the calculations for Biot's modulus, surface heat transfer coefficient and thermal diffusivity. Of course, if the heat capacity and density of the material is known, at these temperatures, it is then possible to calculate thermal conductivity also.

Summary

Thermal data obtained in the temperature range up to 1400°C using the first low temperature device indicated that measurements at high temperatures were feasible. The manner in which the construction of the second device was developed has been covered, setting forth the many problems at each stage of the development and the solution obtained. The device constructed makes possible the measurement of Biot's modulus, surface heat transfer coefficient and thermal diffusivity up to the temperature limit of the insulation material used on the thermocouple. Two sets of thermal data, one at lower temperature and one at high temperature, may be obtained using the same set-up. These measurements may be made in approximately one hour after the furnaces have reached thermal equilibrium.

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W. B. Crandall
W. B. Crandall
Director of Navy Research Project

G. E. Potter
G. E. Potter
Design Engineer

W. G. Lawrence
W. G. Lawrence
Chairman

References

1. "An Unsteady State Method for Measuring Thermal Diffusivity at High Temperatures", Monthly Progress Report No. 183, Ceramic Research Department, New York State College of Ceramics, September 1951.

2. R. D. Potter and N. J. Grant, "Tungsten - Molybdenum Thermocouples," The Iron Age, pp. 65-69, March 31, 1949.